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CENTRAL FILES NUMBER

LIQUID WASTE DISPOSAL

By Smith

Date AUG 31 1948-12-281

Precipitation of Aluminum Nitrate from Redox Waste Solutions.

The method of removal of aluminum from concentrated waste solutions by precipitation as aluminium nitrate has been described in previous reports. This method is applicable to IAW, IIAW, IDW, and all other solutions which contain a high concentration of aluminum salts, especially the nitrate. The IIAW solution has been used in all experiments to date since it was available and since it contained the right amount of activity, namely 50,000 c/min per milliliter as measured on the first stage of the Geiger counter. With this level of activity, samples up to 100 ml. could be handled safely even in the temporary laboratories. This made possible the determination of decontamination factors up to 1000 with reasonable accuracy.

Since the minimum solubility of aluminum nitrate occurs at a nitric acid concentration of 85 grams of HNO_3 per 100 grams of solution, the loss of aluminum will depend upon the correct adjustment of the acidity at the times of precipitation and filtration. This is important since the final concentration of the very active wastes will be greatest if the aluminum is completely removed.

Some other elements such as barium, strontium, and columbium also precipitate under these conditions and it may be necessary to choose the best conditions to obtain a high decontamination factor with a low loss of aluminum nitrate. The best conditions obtainable with the laboratory equipment were a decontamination factor of 2000

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and a recovery of 94% of the aluminum when two cycles were used.

The procedure was tested on a slightly larger scale in order to obtain operating data and to check the design of possible pilot plant equipment. For the first or preliminary run, an inactive solution having the same composition as IIAW was used. This was prepared by dissolving

4 ml. 70% HNO_3

70 grams $\text{Al}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$

in water and diluting to 200 ml. This solution was added to 200 ml. of 70% HNO_3 and distilled. Below are given the vapor and liquid temperatures for various fractions of the distillate.

<u>Volume of Distillate</u>	<u>Vapor Temperature</u>	<u>Liquid Temperature</u>
0		115°
1 drop	108°	118°
10 ml.	114°	119°
30 ml.	115°	119°
50 ml.	116°	120°
60 ml.	116°	120°
80 ml.	117.5°	121°
100 ml.	118°	121.5°
120 ml.	119°	122.5°
140 ml.	120°	123°
160 ml.	120.5°	123.5°
180 ml.	121°	124.5°
200 ml.	121°	125°

The distillate became more and more concentrated in nitric acid until at the last, essentially constant boiling nitric acid was being distilled over. It may be advantageous to use much less nitric acid in this step.

The concentrated solution from the distilling flask was transferred to the precipitator, a vessel similar to the one illustrated. The actual precipitator used in the experiment had the jacket above the glass disc. This caused some difficulty since the heat transfer was not efficient.

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After the solution had been heated to 95° C. by passing steam through the jacket, the aluminum nitrate was precipitated by the addition of 200 ml. of fuming nitric acid. The steam was turned off and cold water slowly run through until the thermocouple read 70° F. The excess nitric acid was removed by suction and the crystals were washed with a small amount of a 1-1 mixture of 70% nitric acid and fuming nitric acid. The air was turned on and the crystals re-dissolved in 150 ml. of 70% HNO₃ at a temperature of approximately 75° C. The mixture was stirred constantly with air and 150 ml. of fuming nitric acid were added to re-precipitate the aluminum nitrate. The steam was shut off and the mixture cooled by allowing the cold water to flow slowly through the jacket. The nitric acid was filtered off with suction as before. Finally the aluminum nitrate was re-dissolved in a minimum amount of hot water with air stirring and removed by suction. These experiments showed that it was possible to use this apparatus for several successive precipitations of the aluminum nitrate.

The concentrated nitric acid which was separated from the aluminum nitrate crystals by filtration was distilled. The temperatures of the vapor and liquid were noted and are given below for different fractions of the distillate.

<u>Volume of Distillate</u>	<u>Vapor Temperature</u>	<u>Liquid Temperature</u>
0	95° C.	115° C.
10	108	117
20	110	119
30	113	119
50	114	120
90	116	121
120	118	122
150	119	123
200	120	124
250	121	124
290	122	125
320	122	124
360	123	124

[REDACTED]

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The first 168 ml. of distillate were analyzed by the Redox Analytical Group and found to contain 87.5 grams of nitric acid per 100 grams of solution. The last fraction contained 73 grams of nitric acid per 100 grams of solution.

It will be advantageous to have not only the total decontamination but also the specific decontamination factors for the main constituents. These are probably Zr-Cb, Ce, Ru, and to a lesser extent Cs and Sr. The Redox Analytical Group, in connection with the research on analytical methods for ruthenium, analyzed the various fractions for ruthenium. The results are not complete but they indicate that ruthenium does not concentrated in any part of the process.

During the next month the large scale apparatus will be used for checking the procedure using as feed solutions IIAW, IDW, and diluted IAW. As far as possible the yields and specific and total decontamination factors will be determined.

The procedure is very arbitrary at present since not enough experimentation has been done to determine the best conditions. Some study should be done on the effect of rate of precipitation of the aluminum nitrate.

The nitric acid solution from the second precipitation will contain only small amounts of impurities and fission products. It is probable that the concentrate from this solution could be added to the feed for the next cycle. These and other problems will be studied as time permits.

A description of these tests and the conclusions reached were written as a memo.